

LABNOTES *Spring 2002*



Labs of the Year Recognized

The Laboratory Certification Program recently recognized environmental laboratories in Marinette and Cadott as the winners of the 2002 Registered Laboratory of the Year Award. The award for large registered facilities went to TSS-Ansul, Inc.'s environmental control laboratory in Marinette, while the award for small registered facilities went to the Village of Cadott wastewater treatment plant. The awards were presented during the Natural Resources Board meeting on March 27, 2002. This is the seventh year the Department has presented the awards.

"The work of environmental laboratories provides the foundation for the science-based decisions that shape departmental policy," said David Webb, manager of the agency's Laboratory Certification and Registration Program. The efforts and expertise of these environmental laboratories plays a critical – but sometimes overlooked – role in the protection of Wisconsin's environment and its residents' health.



ESS Chief David Webb poses with the 2002 Lab of the Year recipients. (left to right) Mr. Dan Burns, Cadott WWTP; David Webb; and Mr. George Rogers and Ms. Judy Tost, TSS-Ansul Inc.

This year's honored labs demonstrated excellence in the field of environmental analysis as well as dedication to generating high-quality chemical data. They were also recognized for surpassing the

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Labs of the Year, continued.

minimum baseline requirements necessary to maintain compliance with department regulations.

The TSS-Ansul laboratory provides analytical support for the company's fire protection product plant. In addition to performing a wide range of tests on the plant's wastewater discharge stream, the lab also performs routine testing of its groundwater monitoring wells.

When nominating the Ansul laboratory for its award, DNR Regional Certification Coordinator Rick Mealy noted how impressed he was with the level of effort invested by laboratory staff to assure the high quality of the results of the arsenic testing performed on the plant's outfall. Mealy also made special mention of the thorough action taken to correct problems when they are identified by the laboratory's quality control measures.

The Village of Cadott wastewater treatment lab provides analytical support for the village's wastewater treatment plant and analyzes wastewater samples for biological oxygen demand and total suspended solids. West Central Region Wastewater Engineer Steve Thon and Regional Certification Officer Camille Johnson nominated the laboratory for the award. In their nomination, Thon and Johnson highlighted the laboratory's practices of analyzing quality control samples at a much higher rate than required by Department regulations and holding the results of these analyses to very strict standards for acceptance. The laboratory uses these data to continually improve the quality of the laboratory operation.

The DNR certifies and registers more than 280 small municipal laboratories, 70 industrial laboratories, and 11 public health laboratories. It also certifies approximately 90 commercial laboratories.

Contact Greg Pils at (608) 267-9564 or by e-mail at pilsg@dnr.state.wi.us for more information. □

LabNotes

Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

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This publication is available in alternative format (large print, Braille, audio tape, etc.) upon request. Please call (608) 267-7633 for more information.

This document is available electronically at www.dnr.state.wi.us/org/es/science/lc.

This document is intended solely as guidance and does not include any mandatory requirements except where requirements found in statute or administrative rule are referenced. This guidance does not establish or affect legal rights or obligations and is not finally determinative of any of the issues addressed. This guidance does not create any rights enforceable by any party in litigation with the State of Wisconsin or the Department of Natural Resources. Any regulatory decisions made by the Department of Natural Resources in any manner addressed by this guidance will be made by applying the governing statutes and administrative rules to the relevant facts.



LabNews

Low-Level Mercury Approvals

The Department recently granted approval to two additional laboratories for low-level mercury analysis under the emerging technology provision in s. NR 149.12 (2), Wis. Adm. Code. Both of the labs, En Chem, Inc. – Madison and North Shore Analytical – Duluth, MN, will employ EPA Method 1631.

The two labs bring the total number of Wisconsin-certified facilities with low-level mercury capability to 12. See the low-level mercury page on the Lab Cert. web site to view the entire list of labs, including methods and detection limits in various matrices. □

www.dnr.state.wi.us/org/es/science/lc/info/Hg_low.htm

Tax Delinquency Check for Renewal

Section 299.07, Wis. Stats., requires the Lab Cert. Program to deny certification or registration to laboratories that are delinquent in paying Wisconsin taxes. The Program will be checking the Federal Employer Identification Numbers (FEIN) of all laboratories against a Wisconsin Department of Revenue tax delinquency database prior to renewal this August. Your laboratory may be contacted in June 2002 with a form requesting your laboratory's FEIN. Please respond promptly to avoid a delay in issuing a new certificate. □

NELAP Update

Approximately 200 new laboratories have been accredited through National Environmental Laboratory Accreditation Program (NELAP) accrediting authorities since fall 2001, when we last had a NELAP update in this space. This brings the total number of labs accredited under NELAP to 1,200 facilities in 38 states and territories, and three foreign countries. Four Wisconsin Laboratories are NELAP accredited. For a complete list of NELAP labs, check the web site link below.

No new states have been granted NELAP accrediting authority in the past year. Twelve state agencies in eleven states have adopted the NELAP standards (two agencies in Louisiana participate). □

www.epa.gov/ttn/nelap/accreditlabs.html

Annual Conferences, Meetings

NELAC 8th Annual Meeting

The 8th Annual National Environmental Laboratory Accreditation Conference (NELAC) will be held in Tampa, Florida July 7-12, 2002 at the Wyndham Harbor Island Hotel. For more information see the link below. □

www.treeo.ufl.edu/nelac

WTQA 2002 Symposium

The 18th Annual Waste Testing & Quality Assurance Symposium will be held August 10-15, 2002 at the Hilton Crystal City Hotel in Arlington, Virginia. The symposium is sponsored by the Independent Laboratories Institute (ILI) under a cooperative agreement with the EPA. Check the WTQA web site for more information. □

www.wtqa.org

EPA 12th Annual QA Conference

US EPA Region 6, is holding its 12th Annual Quality Assurance Conference in Dallas, Texas, September 9-12, 2002. For more information contact Charles Ritchey of EPA at (214) 665-8350 or by e-mail at ritchey.charles@epa.gov. □

WWA/AWWA Annual Conference

Wisconsin Water Association (formerly AWWA WS) holds its annual conference September 9-11, 2002 in Stevens Point. Call Jack Albrechtson (608) 831-6554 for details. □

WEFTEC 2002 75th Annual Conference

The 75th Annual Water Environment Federation's Technical Exhibition and Conference is September 28-October 2, 2002 in Chicago, Illinois. Contact Tom Sigmund (414) 847-0568 of Central States Water Environment Federation - Wisconsin Section for information or check the WEFTEC web site link below. □

www.weftec.org

WWOA Annual Conference

The Wisconsin Wastewater Operators' Association Annual Conference will be held October 8-11, 2002 in LaCrosse. Check the WWOA web site for details. □

www.wwoa.org

Program Administration

Fiscal Year 2003 Budget and Fees

The Natural Resources Board unanimously approved the Laboratory Certification Program's fiscal year (FY) 2003 fee schedule at its March 27 meeting. Effective July 1, the cost per relative value unit (RVU) will increase from \$47.00 to \$48.50. FY 2003 fees will increase 3% to \$679.00 for the typical wastewater treatment lab (registered lab base fee + categories 1-4) and \$2861.50 for the typical commercial lab (certified lab base fee + categories 1-8, 10, 12, & 14-16). Individual fee items and the corresponding FY 2003 unit price are listed in the table below at right.

Bills will be mailed to all facilities in May and payment will be due in full by June 30, 2002. Late fees will be assessed to laboratories that fail to pay renewal fees by the established deadline.

For more information about how fees are determined, consult s. NR 149.05, Wis. Adm. Code, which is available on-line at the link below. Please contact Greg Pils at (608) 267-9564 or by e-mail at pilsg@dnr.state.wi.us if you have any fee-related questions. □

www.legis.state.wi.us/rsb/code/index.html.

New Certification Officer for Northern Region

Wastewater laboratories in DNR's Northern Region will be seeing a new face for their next lab evaluation. Susan Watson is stepping out of the Laboratory Certification program to devote more time to writing wastewater discharge permits. Bill Jaeger is now transitioning into the role of Northern Region Certification Officer.

Bill has considerable experience in Wisconsin's water quality program. He started with the DNR in 1974 at Rhinelander as a limited-term water quality biologist and in 1975 moved to Madison as DNR's biologist for reviewing impacts of steam electric generating plants on surface waters.

In 1976, Bill took the opportunity to return to Rhinelander as a water quality field biologist, this time as a full-time employee. Since then he has worked in various activities in the water quality program, including; monitoring and water quality classification of surface waters, nonpoint

pollutant planning and evaluation, oversight of wastewater sludge spreading, revision of water quality standards, calculation of wastewater effluent limits and coordination of Northern Region's newest river protection programs.

Although college was a long time ago, Bill does have a degree in aquatic chemistry and biology. Welcome to the Lab Cert. program! Contact Information for Bill Jaeger is below. □

Mailing Address:

Wi. Dept. of Natural Resources
107 Sutliff Ave.
Rhinelander, Wi. 54501

Phone: (715)365-8971

Fax: (715)365-8932

Email: jaegew@dnr.state.wi.us

Laboratory Fees for FY 2003 **(Sept. 1, 2002 - Aug. 30, 2003)**

Fee Item	FY 2003 Unit Price
Registered Base Fee	\$485.00
Certified Base Fee	\$727.50
Reciprocity Fee	\$1,455.00
Initial Application Fee	\$291.00
Revised Application Fee	\$145.50
Category 1	\$48.50
Category 2	\$48.50
Category 3	\$48.50
Category 4	\$48.50
Category 5	\$97.00
Category 6	\$97.00
Category 7	\$194.00
Category 8	\$194.00
Category 9	\$194.00
Category 10	\$194.00
Category 11	\$194.00
Category 12	\$194.00
Category 13	\$194.00
Category 14	\$194.00
Category 15	\$582.00
Category 16	\$194.00
Category 17	\$582.00
Category 18	\$970.00
Category 18a (Nitrate Only)	\$97.00
Category 18b (Nitrate & Fluoride)	\$194.00
Category 19	\$194.00
Category 20	\$1,261.00
Category 21	\$194.00

NR 149 Revision Update

By David Webb

The advisory committee charged with assisting the Department in updating Chapter NR 149 of the Wisconsin Administrative Code—the administrative rules that govern laboratory certification and registration in Wisconsin—has met three times. After spending its first two meetings laying the foundation for committee operations and discussing roles and responsibilities, deliberation of the content and future direction for the code revisions began in earnest at its last meeting.

Membership on the committee consists of excellent people with valuable input and links to their larger constituencies. We're encouraging laboratories and those interested in laboratory certification and registration to work through their "representative" on the committee to ensure that as many ideas and levels of input are brought to bear as possible. If you're unsure about with whom you should be communicating to have your voice heard, check the list of advisory committee members (see contact list opposite) or contact the Lab Cert. program for help (608/267-7633).

There is also an extensive and growing amount of material on our web site related to the NR 149 revision effort as well as more general information on the laboratory certification and registration program. Please make use of this resource if you're so inclined. The web site address is www.dnr.state.wi.us/org/es/science/lc. The NR 149 button on the left-hand sidebar will take you to the NR 149 Revisions home page. The web site contains agendas and handouts for upcoming meetings, minutes and work products for past meetings, and a running list of code topics and revision ideas currently under consideration by the committee.

In future meetings, the committee will continue to discuss issues ranging from the over-arching, big-ticket items to technical minutia; all of which are important since as we know, the devil's in the details. There will certainly be times when reaching decisions on final revised code language will be very challenging given the myriad pros, cons, and viewpoints to many of the issues. The bottom line will be to arrive at a new code that is fair, efficiently implemented

(affecting costs of administering the program), and assuring data quality to the extent possible. After all, it's data quality that allows effective administration of Department programs and sound science-based decision making.

I also want to thank those on the committee for willingly volunteering their time for this effort. We realize everyone's quite busy and that this involvement is an extra item in already full schedules. We are excited to continue this effort leading to a new code that should result in an overall improved program. □

Advisory Committee Member	Represented Constituency
George Bowman WSLH (608) 224-6278	State Laboratory of Hygiene
Debbie Cawley Green Bay Metropolitan Sewerage Dist. (920) 438-1073	Large Municipal Wastewater Plant
Joe Celmer Little Rapids- Shawano Mill (715) 526-2181	Paper Council
Randy Herwig City of Lodi (608) 592-3247	Small Municipal Wastewater Plant
Paul Junio TestAmerica, Inc.- Watertown (920) 261-1660	Commercial Laboratory
David Kollakowsky Wisconsin Electric Power Company (414) 221-2835	Industrial Laboratory
R.T. Krueger Northern Lakes Service (715) 478-2777	Wisconsin Environmental Laboratory Association
Marcia A. Kuehl MAKUEHL, Company (920) 469-9113	Demonstrated Interest in Laboratory Certification
Ruth Klee Marx County of Marathon Health Department (715) 842-7891	Public Water Utility
Steven Smith BT ² , Inc. (608) 224-2830, ext. 239	Non-Laboratory Data User
Steven Sobek WI DATCP (608) 267-3500	Dept. of Agriculture, Trade and Consumer Protection
Randy Thater Waukesha Wastewater Treatment Plant (262) 524-3627	Municipal Environmental Group

MN-WI Reciprocity Agreement Terminated

By David Webb

The reciprocity agreement for laboratory certification between the states of Minnesota and Wisconsin that had been in place since 1990 was recently mutually terminated. The termination agreement was signed on April 16th, with an effective date of May 16 (30 days post-signing).

Many discussions took place between staff and management of the two state certification programs in an effort to revise the decade-old agreement. A very positive relationship was established, but with the joint realization that the two programs were simply not equivalent enough to warrant retaining the agreement. Also, both states are in the process of making significant revisions to the rules governing each program.

Therefore, it made the most sense to terminate the existing agreement, let each state's rules take shape over the next one to three years, and then re-visit an agreement of some kind between our two states.

Terminating the agreement was a very difficult decision for both programs since we share so many environmental and economic resources. However, it was the right thing to do given the circumstances. My counterpart in Minnesota is as excited as I am in further strengthening our relationship, sharing expertise, and maximizing program efficiency.

Below is the text of a letter that was sent on April 24 to all certified labs in each state. Any lab directly impacted by the termination also received a customized letter in addition to telephone confirmation. □



State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

Scott McCallum, Governor
Darrell Bazzell, Secretary

101 S. Webster St.
Box 7921
Madison, Wisconsin 53707-7921
Telephone 608-266-2621
FAX 608-267-3579
TTY 608-267-6897

April 24, 2002

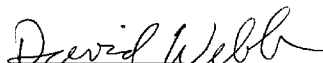
Subject: Termination of Certification Reciprocity Agreement


Dear Certified Laboratories in Minnesota and Wisconsin:

The agreement between the States of Minnesota and Wisconsin that covers environmental laboratory certifications is being terminated. This letter is to inform you in a timely manner in the event that your laboratory has any detrimental impacts from the termination of the reciprocity agreement. This is a mutual decision by our two certification programs. Moreover, our two programs are communicating openly and constructively as our respective programs evolve. Both programs anticipate rules revisions and policy improvements during the next few years. We welcome your suggestions for proposed changes in rules and policies.

We want to assure you that, while our two programs have agreed to terminate the reciprocity agreement, we continue to explore ways to coordinate particular aspects of our programs, as appropriate. We appreciate that our two states share many environmental and economic resources. We understand the importance of working together on certification issues. If your facility is directly impacted by the termination of the agreement, we will be in contact with you under separate cover.

Sincerely,


David Webb, Chief
Environmental Science Services
Wisconsin Dept. of Natural Resources


Louise W. Liao, Ph.D.
Environmental Lab Manager
Minnesota Dept. of Health

Quality Matters - Qualifiers

By Donalea Dinsmore, DNR QA Coordinator

If you had to choose between sample results that are qualified and those that aren't, which would you choose? Those of us in the business of reviewing environmental data understand that qualified data isn't necessarily "bad" or unusable data. Sometimes, results that are qualified are of a higher quality than data that is not. The assessment of usability depends on the laboratory's system for qualifying results, the nature of the qualifier, the intended use of the data, or the knowledge of the person reviewing the data.

The US EPA contract laboratory program (CLP) provides a model for a set of qualifiers that function effectively because it is a component of an entire reporting system that is well-defined and understood by those using it. The system relies on a limited list of flags: B - blank contamination, U - undetected, R - rejected or unusable, N - non-target analyte, Q - QC failure, and J - estimated result. Although there are multiple reasons for assigning any particular code, they serve as cautions to data users to look for additional information. Several rules, conventions, and assumptions support the system so the qualifiers aren't used in isolation. Contracts or project plans contain explicit data acceptance criteria, data is validated against the project objective using an established protocol, and the person validating the data prepares a case narrative that provides relevant information for interpreting the data, which is available to those using the data.

For better or worse, the DNR's present systems for generating, reporting and assessing compliance data aren't as regimented as CLP. The laboratory certification code includes general requirements that laboratories qualify data under certain circumstances, but doesn't dictate specific language for qualifiers or conventions for their use so laboratories develop their own. Data qualifiers may be ambiguous or contain minimal information about the "problem" so the client or regulator may not be able to determine the data quality without a case narrative or contacting the laboratory. Or, the text for the qualifier may be lengthy, providing specific information to aid in interpreting the results. One laboratory may qualify a result, indicating that the analysis

exceeded the regulatory holding time while another laboratory's convention is to report the sample collection and analysis dates and let the data user determine whether the elapsed time between collection and analysis is a problem. To add to the confusion, reporting systems for compliance data may not be designed to capture descriptive qualifiers and case narratives. Even when data qualifiers are part of reporting, all too often, qualifiers don't follow sample results through reporting systems and into regulatory databases, increasing the potential that results will be misinterpreted. There's got to be a better way!

As the Quality Forum discusses how to establish a standard set of data qualifiers that function effectively, we are considering how much information should be incorporated into a qualifier and what changes to certification rules and reporting systems will affect when and how data is qualified. For instance, if our rules specify that DNR will not accept sample results unless the calibration and all associated calibration checks pass specified acceptance criteria for each reported analyte, then we shouldn't need qualifiers for calibration failures. Conversely, if the rules allow a certain number of analytes to fail before considering the analysis to be out of control, then we need to establish whether any results get qualified and conventions for doing so. If our reporting systems are changed to check for holding time exceedances when the data is received at DNR, should laboratories continue attaching this qualifier to results?

The Quality Forum is an ad hoc group of lab professionals who discuss issues of mutual concern to DNR programs and laboratories (see article in LabNotes Fall 2001). The discussions at Quality Forum meetings have been lively. We are meeting at six-week intervals and have had three sessions so far. I extend special thanks to the laboratory representatives that have volunteered their time: James Chang, APL; Bill Hughes, Madison Metropolitan Sewage District; Paul Junio, TestAmerica; Art Lautenbach, EnChem; Matt Roach, State Lab of Hygiene; Dale Piechocki, Environmental Health Laboratories; Jim Salkowski, US Filter; Randy Thater, Waukesha Wastewater Treatment Plant and Amy Tutwiler, Municipal Environmental Group. For more information on the Quality Forum, contact Donalea Dinsmore at dinsmd@dnr.state.wi.us or (608) 266-8948. □

Council Corner

By Paul Junio, Council Chair

I wish a warm welcome to all of you reading this. I am the current Chair of the Certification Standards Review Council, a statutory council that advises the DNR Laboratory Certification and Registration program. The Council, in its current incarnation, is due for a shake-up later this year, as two appointees will be finishing their second and final terms. Dave Kolakowsky, representing Industrial Laboratories, and Ruth Klee Marx, representing Public Water Utilities, have both arrived at mandatory retirement due to having served two consecutive three-year terms. Their input on the Council will be missed.

The departure of Dave and Ruth means that the Council is looking for appropriate replacements for them. Also of note are the continued openings for Solid and Hazardous Waste Disposal Facility and Agricultural Interests (defined by statute as a farmer actively engaged in livestock production). Anyone who represents any of these constituencies and is interested in serving on the Council should contact Phillip Spranger at (608) 267-7633 or by e-mail at spranp@dnr.state.wi.us for information on the nomination process.

Much of the Council's activities of late have been focused on the re-write of chapter NR 149, Wis. Adm. Code. The Council serves as the core of the NR 149 Revision Advisory Committee that has been meeting on a regular basis since the beginning of the year on this issue. Meetings at approximately six-week intervals are scheduled through the end of this year. Any input on the Certification Code can be forwarded to any Committee member. Or, if you're the outgoing type, feel free to join us at one of our meetings. Meeting minutes and schedules are posted on the Lab Cert. web site (see link below). □

www.dnr.state.wi.us/org/es/science/lc/nr149

A Statutory Council

The Secretary of the Department of Administration appoints eight of the Certification Standards Review Council members as follows:

- One member to represent municipalities having wastewater treatment plants with average flows of more than 5,000,000

gallons per day.

- One member to represent municipalities having wastewater treatment plants with average flows of less than 5,000,000 gallons per day.
- One member to represent industrial laboratories with permits issued under ch. 283.
- One member to represent commercial laboratories.
- One member to represent public water utilities.
- One member to represent solid and hazardous waste disposal facilities.
- One member with a demonstrated interest in laboratory certification.
- One member who is a farmer actively engaged in livestock production to represent agricultural interests.

The chancellor of the University of Wisconsin-Madison appoints one member to represent the state laboratory of hygiene. □

Council Contact List

Agricultural Interests

Vacant

Commercial Laboratories

Mr. Paul Junio - (920) 261-1660

pjunio@testamericainc.com

Demonstrated Interest

Ms. Marcia Kuehl - (920) 469-9113

makuehl@aol.com

Industrial Laboratories

Mr. David Kollakowsky - (414) 221-2835

dave.kollakowsky@wepco.com

Municipal Wastewater Plant (> 5 mgd)

Ms. Debbie Cawley - (920) 438-1073

dcawley@gbmsd.org

Municipal Wastewater Plant (< 5 mgd)

Mr. Randy Herwig - (608) 592-3247

rherwig@wppisys.org

Public Water Utilities

Ms. Ruth Klee Marx - (715) 842-7891

rkm Marx@mail.co.marathon.wi.us

Solid and Hazardous Waste Disposal Facility

Vacant

State Laboratory of Hygiene

Mr. George Bowman - (608) 224-6278

gtb@mail.slh.wisc.edu

Proficiency Testing

Disinfection By-products PT Providers

Several Wisconsin-approved reference sample providers received approval to also provide reference samples for the Safe Drinking Water Act disinfectant and disinfection byproduct (DBP) analytes bromate, chlorite and haloacetic acids (five). The regulated haloacetic acids are monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, bromoacetic acid and dibromoacetic acid.

One of the requirements for laboratories to become certified to perform DBP testing for Wisconsin clients is the successful analysis of DBP reference samples (or PTs). An HAA5 PT must contain all five of the regulated haloacetic acids and labs must obtain acceptable results on at least four of the five (80%).

Existing providers with National Institute for Standards and Testing (NIST) approval for DBPs were granted approval to provide those PT samples for Wisconsin clients. The Approved Reference Sample Providers list on the Lab Cert. web site has been updated with this information. (See related article on page 13.). □

ASI Approved for Categories 01-06

Analytical Standards, Inc. (ASI) has completed the process of becoming a Wisconsin-approved reference sample provider. The approval is for tests in test categories 01 through 06 only.

Check the Lab Cert. web site for more information. ASI's contact information is:

Analytical Standards, Inc.
6331 Emerson Avenue
P.O. Box 4060
Parkersburg, WV 26104-4060
Phone: (8000) 283-4844
Web Site: mcnet.marietta.edu/~asi/

WSLH Discontinues Organics PTs

As reported here in LabNotes Fall 2001, the Wisconsin State Laboratory of Hygiene has discontinued all organic proficiency testing samples. The WSLH blind standards program was also affected.

The WSLH continues to offer inorganic PT studies three times a year for both water pollution

(WP) and water supply (WS) tests to meet Wisconsin certification requirements and for blind standards to meet quality assurance needs. The program will also continue to provide water microbiology samples to meet certification requirements for ATCP 77 and to meet quality assurance needs. □

DMRQA-22 Update

EPA mailed Discharge Monitoring Report - Quality Assurance (DMR-QA) Study 22 announcements to affected facilities in mid-May. The start date for Study 22 is June 5, 2002 and the end date is September 20, 2002. Because of the timing, "regular" DMR-QA PT studies cannot be used for Wisconsin renewal. Graded reference sample results for Wisconsin renewal must be received by the Lab Cert. program no later than August 31, 2002; regular DMR-QA study results won't be released until after the September 20, 2002 close date.

On page 5 of the Study 22 instructions is a note on using an "approved" water pollution PT study instead of a DMR-QA study to meet both DMR-QA and state certification requirements. However, for Wisconsin certification or registration renewal, the requirement to participate in a PT study for which final graded results are released after June 5 really leaves the laboratory little time to correct unacceptable results prior to Wisconsin's renewal deadline.

Laboratories should consider analyzing reference samples from a Wisconsin-approved provider as soon as possible each year to make sure all Wisconsin Laboratory Certification or Registration renewal requirements are met well in advance of the August renewal deadline. This leaves time for a repeat study to correct any unacceptable results.

EPA intends to include the whole effluent toxicity (WET) test requirement in Study 22. However, a WET test proficiency testing provider had not been approved prior to the DMR-QA mailing. EPA will contact labs in July with more information on the WET component.

Questions about DMR-QA should be directed to Phillip Spranger at (608) 267-7633 or at spranp@dnr.state.wi.us. □

Wastewater Focus

Pulp and Paper Cluster Rule Q&A

Implementation of the EPA “Pulp and Paper Cluster Rule” has raised questions for permitted facilities and laboratories alike. Here’s a question and answer that bears repeating.

Q: “My Wisconsin Pollution Discharge Elimination System (WPDES) permit does not include a discharge limit for chemical oxygen demand (COD) or total organic carbon (TOC), but the permit requires monitoring of process stream liquid for COD or TOC to assess the effectiveness of the facility’s best management practices (BMP) for spent pulping liquor management, spill prevention and control. Do these COD or TOC tests need to be performed by a certified lab?”

A: Yes. A lab certified or registered must perform these analyses. Monitoring required by terms and conditions of a permit issued under ch. 283, Wis. Stats., must be performed by a laboratory certified or registered under ch. NR 149, Wis. Adm. Code., unless the test is specifically excluded in s. NR 219.06, Wis. Adm. Code. The tests excluded from the certification or registration requirement are temperature, turbidity, bacteria tests in wastewater effluent and sludges, pH, chlorine residual, specific conductance, physical properties of soils and sludges, nutrient tests of soils and sludges, and flow measurements. □

BOD Holding Time Clarified

The enforceable holding time for biochemical oxygen demand (BOD) samples is 48 hours from the end of the composite period. This issue has caused some confusion because method and law both recommend performing the analysis as quickly as possible.

Standard Methods is clear that the holding time for BOD samples is 24 hours post composite period. However, ch. NR 149, Wis. Adm. Code also requires adherence to applicable state or federal law and ch. NR 219, Wis. Adm. Code allows a 48 hour post composite period holding time. In this case, state law (ch NR 219) is the driving factor.

Emphasis on sample preservation is in order here—if samples are faithfully cooled to 4 C or less, then degradation should be minimized. □

Super-saturation (BOD)

One of the most common BOD-related deficiencies identified in laboratories is that samples appear to be supersaturated with dissolved oxygen (DO). The key to identifying supersaturation is to know the saturation point (for oxygen in water) on each day of analysis. Standard Methods directs analysts to bring their dilution water to saturation before use. Perhaps what is implied but not clearly stated is the importance of performing an accurate calibration, again based on a sample of saturated (dilution) water.

The average elevation in Wisconsin is about 800 to 1000 feet above mean sea level. This means that normal pressure is about 28.94 inches of mercury (735 millibars). Using a chart of oxygen saturation based on temperature and pressure, the saturation point for a lab at 735 millibars pressure and 20°C is 8.76 mg/L. Therefore, any water sample that is found to contain more than 8.76 mg/L DO at this temperature and pressure is supersaturated. (Note as well that the saturation point decreases as temperature increases.) It simply is not possible for samples under these conditions to hold more DO without being super-saturated. Your auditor will determine the oxygen saturation point for a given day’s analyses and then scan sample initial DO readings for values greater than the saturation point.

If supersaturation is a problem in your laboratory, first evaluate your calibration technique. Improper calibration accounts for a significant percentage of problems that appear to be supersaturated samples. In addition, shake samples (i.e., bring them to saturation) once they are at room temperature—not before. Shaking samples while they are still cold will cause supersaturation. Look at the shaking procedure not as a means of ridding samples of supersaturation, but as a means of bring the oxygen content to the saturation point. □

BOD calibration technique.

Another frequently identified BOD-related deficiency relates to poor calibration technique. Most laboratories calibrate their DO meters by placing their probe in a DO bottle that contains only a small amount of water. The probe never touches the water. This technique is termed the “water-saturated air” method of calibration, and most manufacturers recommend it. Does it work? Absolutely. But the “catch” is that for optimal results, the air inside the BOD calibration bottle must be 100% humidity (i.e. air saturated with water). Is that the case in your laboratory?

What is the first step virtually every lab analyst does (and should do) before calibrating the DO meter? The answer is that they remove the DO probe from the calibration bottle and either shake off or wipe off any water (condensation that has formed) on the probe membrane. Once that probe is taken out of the bottle, however, what happens to the “100% humidity” environment (assuming it was to begin with)? That’s right...the bottle humidity very quickly equilibrates with the humidity inside the laboratory. The end result is that the pre-requisite for proper calibration—water-saturated air—is generally not satisfied, leading to calibration problems.

To better highlight this problem, consider the way in which DO is measured during calibration (a bottle filled with air) as compared to routine sample measurement (bottle filled with water). Using an old analogy, this amounts to comparing apples and oranges. It is important at this point to emphasize once again that the water-saturated air method of calibration can and does work—provided the bottle contains water-saturated air. The theory is that in placing the probe back into the calibration bottle, the air in the bottle reaches equilibrium once again (becomes water-saturated) very quickly (in minutes). In fact, creating a 100% humidity environment (even on this small scale) is bound by the laws of physics, and saturation cannot be achieved passively in a few minutes time. The bottom line is that calibration is performed without an environment of water-saturated air, introducing some degree of error to the analysis—enough error to cause blank failures.

If you are having trouble with super-saturation, or you find that your blanks frequently

appear to gain oxygen, consider switching from the water-saturated air to the air-saturated water method of calibration. If you choose to make this switch, however, be aware that some probes are equipped with an “air” vs. “water” calibration switch. Be sure that the switch reflects the correct calibration medium. □

"Fast-attack" method for pouring out TSS and BOD samples.

A number of QC failures of either BOD or TSS replicates can be traced to the technique used by the analyst to measure the volume used for sample analysis. One of the basic rules of laboratory analysis is to make very precise measurements. Consequently, when pouring out samples for either BOD or TSS, the analyst may pour the sample very quickly initially, but as the desired mark (e.g., 250 mLs) approaches, the pouring operation slows down considerably. The intent is to reach the [250 mL] mark without going “over the line”. In the worst case scenario, the analyst may even exceed the desired mark, in which case he/she slowly pours some of the sample back into the original sample bottle.

To resolve this problem, operators and analysts first need to “unlearn” their analytical techniques. This is perhaps the only instance in which being careful and precise does more harm than good. Even if the sample was thoroughly mixed immediately before the aliquotting process begins, during the entire time it takes to measure out the desired sample any solids in the sample have already begun to settle out. Clearly, the longer it takes to produce an exact target volume, the more that heavier solids have settled, and the analyst can, in some cases, be pouring supernatant rather than true sample. If the same sample container is then used to measure out sample volume for a replicate, it should also be clear that a greater proportion of solids exists in the remaining sample volume. This can—and frequently does—account for significant discrepancies between replicates. The auditor will also see it translate into excessively broad control limits.

The best technique for pouring out samples for BOD and TSS (and any other samples that contain significant concentrations of suspended

Continued on page 12.

Fast Attack, continued.

material) is to adopt a “fast attack” procedure for pouring out sample volumes. Rather than focusing on a specific desired target volume (e.g., 250 mL), mix the sample well and then pour as quickly as you can with the goal of getting close to your target volume without exceeding the mark. You will have a far more representative sample if you quickly pour out 243 mLs of sample into a graduated cylinder than if you take the extra time necessary to obtain exactly 250 mLs. More importantly, the remainder of the sample will be equally representative of the true sample, and better performance on replicates (leading to better control limits) will result.

The only drawback to this approach is that you may need to use a calculator to determine final sample concentration where an exact sample volume could allow you to do the math quickly, in your head. □

Got carbonaceous BOD?

We are quickly approaching the annual certification/registration renewal period. This means that late in August laboratories will be receiving an updated certificate documenting their certifications or registrations. Laboratories that submit applications during the course of a certification period may also get a new certificate during the fiscal year once they have satisfied the application and evaluation process. Any time a new certificate regarding registration/certification is received, laboratories should be checking the certificate for accuracy.

How should laboratories check their certificates? Facilities with a WPDES permit should check their certificates against the analytical procedures performed in their laboratory for compliance with their permit requirements. Any parameter required to comply with permit requirements must appear on your certificate. The only exceptions are pH, residual chlorine, and fecal coliforms. Other laboratories should verify that each parameter for which results are submitted for a covered program (see note under s. NR 149.02, Wis. Adm. Code) appears on their certificate.

Performing tests for compliance with a covered program, such as a discharge permit, for parameters that do not appear on your certificate

represents a violation of the Laboratory Certification and Registration Code (NR 149), and generally will also be a violation of other Codes associated with the covered program involved. A number of wastewater facilities have had their permit monitoring requirements changed from BOD to carbonaceous BOD without taking the proper steps to add CBOD to their laboratory certificate.

If you find that your certificate does not accurately reflect testing you perform for a covered program, please contact Phillip Spranger at (608) 267-7633 or spranp@dnr.state.wi.us. If there is an error on our part, the correction can be made quickly and a new certificate will be issued. Otherwise, a revised application, with applicable fees and any reference sample requirements will have to be submitted to resolve the discrepancy. □

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Drinking Water

Disinfection Byproduct Certification

Effective January 1, 2002, federal and state drinking water regulations require certain public water suppliers to monitor for the disinfection byproducts (DBPs) bromate, chlorite and haloacetic acids (five). The five regulated haloacetic acids (HAA5) are monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, bromoacetic acid and dibromoacetic acid. Laboratories performing analyses in support of DBP monitoring must be certified under ch. NR 149, Wis. Adm. Code.

In fall 2001, a letter was sent to all drinking water certified labs explaining the requirements for certification. As of May 2002, five labs had applied for and received certification for some/all of the DBPs.

If your laboratory is interested in becoming certified for DBPs, contact Certification Chemist, Phillip Spranger at (608) 267-7633 or by e-mail at spranp@dnr.state.wi.us. □

UCMR Certification Not Offered by Wisconsin Lab Cert. Program

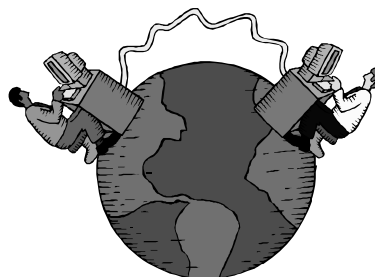
The Wisconsin Laboratory Certification and Registration program will not be offering certification for Safe Drinking Water Act unregulated contaminant monitoring rule (UCMR) analytes. The WDNR drinking water program is not directly implementing the UCMR in Wisconsin and, as a result, the UCMR contaminants were not included in ch. NR 809, Wis. Adm. Code. Since the contaminants are not listed in ch. NR 809 with maximum contaminant levels (MCLs), the Lab Cert. program does not have the authority to certify for the analytes.

The US EPA will be directly implementing the UCMR in Wisconsin and UCMR monitoring results are to be reported directly to EPA. Laboratories that are certified by EPA or by a state with direct responsibility (primacy) for UCMR implementation may perform UCMR analyses for Wisconsin clients.

The EPA may grant approval on a case-by-case basis to Wisconsin laboratories that wish to perform analyses in support of the UCMR if the

laboratory is certified by WDNR for methods "similar" the methods approved for UCMR contaminants. For example: labs Wisconsin-certified for EPA Method 525.2 may obtain EPA approval to perform UCMR analyses using methods 526 and 528 for semivolatiles; labs certified for 549.1 may be approved for 532.

One complication for Wisconsin-certified labs is that the Wisconsin program does not explicitly certify by method (i.e., the Wisconsin certificate does not list methods for which the laboratory is approved). However, since the Wisconsin program does audit for approved methods and does assure that all method-specific requirements are being met, labs certified for SDWA analytes using the approved methods may petition EPA for UCMR approval. □



Test Data Needed for Electronic Data Submittal System

The DNR Bureau of Drinking Water and Groundwater has developed a computer system for receiving monitoring data electronically for compliance with chapters NR 809 and NR 812, Wis. Adm. Code (public water supplies and private wells, respectively). The department met with a focus group on March 9, 2001, to get input on the new system. DNR is looking for laboratories willing to provide monitoring data so the new system can be tested. Contact Ron Arneson at arnesr@dnr.state.wi.us or (608) 264-8949 if your laboratory is interested in providing test data. Check the Laboratory Services web page for more information (see link below). □

www.dnr.state.wi.us/org/es/science/lsl/lab_data

Regulatory Update

Phasing Out Paper TADs

Laboratories may receive inquiries about providing electronic data submittals for groundwater monitoring at landfills. Although the Bureau of Waste Management has required larger facilities to submit data electronically since 1996, some small landfills with fewer than 10 wells were allowed to submit paper turn-around documents (TADs). Beginning July 1, 2002, the Bureau of Waste Management will no longer print paper TADs. Each of the landfills that submits paper TADs received a letter and was contacted by telephone notifying them of the change and reviewing procedures for submitting data electronically.

The Bureau's web page includes information on formatting electronic files, a spreadsheet template and the current list of parameter numbers and descriptions (see link below). □

www.dnr.state.wi.us/org/aw/wm/monitor

Parameter Number Changes

It's unfortunate, but over time some of the parameter numbers for groundwater monitoring at Waste Management Program facilities have changed. If you haven't downloaded the list of parameter numbers recently, we encourage you to check that you have a current list. The file can be downloaded from DNR's web site (see link below). The Bureau of Waste Management is working on a list of parameter numbers that have changed and once completed, that will be available on the web site as well. Many landfill plans of operation and plan modifications include parameter numbers as part of the monitoring schedule. If the number listed in the Plan differs from the list you have, make sure you have the most current parameter list and use the number from that list. If you have questions, please contact Kathy Thompson at (608) 266-0867 or thompk@dnr.state.wi.us or Mary Beth Schuetz at (608) 267-0546 or schuemb@dnr.state.wi.us. □

www.dnr.state.wi.us/org/aw/wm/monitor/downloads/parm_tbl_by_name.pdf

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Substances of Concern at Low Levels

Spring! Time for the Laboratory Certification Program to publish its annual list of substances of concern at low levels. This list is published as a reminder that s. NR 149.15 (3), Wis. Adm. Code, requires labs to report all results greater than the limit of detection (LOD) for those substances with standards specified in chapters NR 105, 140, and 720, Wis. Adm. Code, that are below the laboratory's limit of quantitation (LOQ). All results greater than the LOD but less than the LOQ must be appropriately qualified (consult NR 149 for definitions of the LOD and LOQ).

Chapter NR 809, Wis. Adm. Code, also requires this information to be reported for all regulated primary drinking water contaminants. Be aware that some programs may require laboratories to report the results of all compounds down to the LOD, even if they do not appear on this list. It is the laboratory's responsibility to ensure that reporting requirements are met. Check with your clients or DNR staff to determine what reporting requirements apply. Labs are encouraged to report all results down to the LOD, thereby avoiding confusion and insuring reporting requirements are always met.

A table of regulated substances and the associated standards under Chapters NR 140, 809, and 720, Wis. Adm. Code, is available in Chapter 6 of the "Program Information and Requirements" handbook, (also known as the "Yellowbook"), which can be down loaded from the Program's web site at the link below. □

www.dnr.state.wi.us/org/es/science/lc

INORGANICS

Metals

Antimony
Beryllium
Cadmium
Lead
Thallium
Mercury
Chromium (Hexavalent)

ORGANICS

Acids/Phenols

Pentachlorophenol (PCP)

Benzidines

Benzidine

Haloethers

Bis(chloromethyl)ether

Nitroaromatics

2,4-Dinitrotoluene
2,6-Dinitrotoluene

ORGANICS

Polynuclear Aromatic

Hydrocarbons

Benzo(a)pyrene

Phthalates & Adipates

Di(2-ethylhexyl)phthalate

Nonpurgeable Chlorinated

Hydrocarbons

Hexachlorobenzene

Dioxins/Furans

Dioxin

PCBs

Polychlorinated biphenyls

Chlorinated Pesticides

DDT and Metabolites
Heptachlor
Heptachlor epoxide
Lindane
Toxaphene

ORGANICS

Carbamate Pesticides

Aldicarb

Nitrogen Pesticides

Alachlor
Dimethoate
Parathion
Trifluralin

Volatiles

1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,3-Dichloropropene (cis/trans)
Bromodichloromethane
Bromoform
Bromomethane
Chloroform
Chloromethane
Methyl tert-butyl ether (MTBE)
Methylene Chloride
Vinyl Chloride
Dibromochloropropane (DBCP)
Ethylene dibromide (EDB)



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